



UNIVERSITI PUTRA MALAYSIA

**PREPARATION AND CHARACTERIZATION
OF LITHIUM-BASED SOLID STATE BATTERY MATERIALS**

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**PREPARATION AND CHARACTERIZATION OF LITHIUM-BASED SOLID
STATE BATTERY MATERIALS**

By

MUHAMMAD AMIN IDREES

**Thesis Submitted in Fulfilment of the Requirements for the Degree
of Doctor of Philosophy in the Institute of Advanced Technology
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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirements for the degree of Doctor of Philosophy

**PREPARATION AND CHARACTERIZATION OF LITHIUM-BASED
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Faculty: Institute of Advanced Technology

Three different cathodes such as LiMn_2O_4 , LiCoO_2 and $\text{Li}_2\text{Ni}_8\text{O}_{10}$ were synthesized by the sol-gel technique. The said materials at low temperature were achieved through the low temperature technique. The X-ray diffraction study of the compounds confirmed the formation of single-phase compound at higher calcination temperature. At low temperatures the X-ray diffractogram of the samples showed the presence of low intensity diffraction lines with weak impurities indicating the existence of crystallinity but these were not indexed to any kind of impurities of LiMn_2O_4 , LiCoO_2 and $\text{Li}_2\text{Ni}_8\text{O}_{10}$. The formation temperatures of the compounds were analyzed using DTA. The DTA studies showed clearly the lowest formation temperature and this formation temperature depends upon the gelating agent used in the present study. The lowest formation temperatures recorded were 208 °C for LiMn_2O_4 , 201 °C for LiCoO_2 and 214 °C for $\text{Li}_2\text{Ni}_8\text{O}_{10}$.

The thermogravimetric analysis showed that the compounds were stable up to 800 °C. The EDAX analysis was performed for the compounds to identify the purity of the compounds. The EDAX spectrum showed that there was no impurity present in the compounds. It ascertained the formation of single-phase compounds by XRD. Because of low atomic weight lithium could not be detected other than that the EDAX showed the presence of the respective atoms. The particle size distribution of the compounds showed that the particles were distributed in large volume. The particle diameter increased with the increase of calcination temperature. Grinding reduced the large volume distribution and the particle diameter. After grinding by mortar and pestle hand grinder, the particle size was reduced much and the distribution was narrowed down, thereby the surface area of the particle was increased.

The SEM analysis also confirmed the sub-micron size reduction and the distribution was narrowed down, thereby the surface area of the particle was increased. The compounds were used as electrode materials for lithium ion batteries. The battery analysis showed that the capacities of the LiMn_2O_4 , LiCoO_2 and $\text{Li}_2\text{Ni}_8\text{O}_{10}$ were 10 mAh, 24 mAh and 5 mAh respectively.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
Sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

**PENYEDIAAN DAN PENCIRIAN BAHAN-BAHAN BATERI
KEADAN PEPEJAL LITIMUM**

Oleh

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Tiga jenis bahan katod iaitu LiMn_2O_4 , Li CoO_2 dan $\text{Li}_2\text{Ni}_8\text{O}_{10}$ telah disintesis menggunakan kaedah itu 'sol-gel'. Kaedah suhu rendah ini menghasilkan bahan-bahan yang tersebut di atas pada suhu rendah. Kajian belauan sinar-X terhadap sebatian-sebatian ini mengesahkan pembentukan sebatian fasa tunggal pada suhu 'kalsinasi' yang lebih tinggi. Pada suhu rendah difraktogram sinar-X sampel-sampel menunjukkan kewujudan puncak-puncak belauan yang kecil beserta kehadiran sedikit bendasing, menandakan kewujudan sifat kehabluran, tetapi tidak pula merujuk kepada sebarang ketidaktulenan LiMn_2O_4 teroksida. Suhu pembentukan sebatian tersebut dianalisa menggunakan kajian DTA menunjukkan dengan jelas suhu pembentukan terendah ini dan suhu pembentukan ini bergantung kepada 'gelating agent' yang digunakan didalam penyelidikan ini. Suhu pembentukan terendah yang direkodkan adalah 208°C bagi LiMn_2O_4 , 201°C bagi Li CoO_2 dan 214°C bagi $\text{Li}_2\text{Ni}_8\text{O}_{10}$.

Analisa termogravimetrik menunjukkan sebatian-sebatian tersebut adalah stabil sehingga 800°C. Analisa EDAX dilakukan untuk mengenalpasti ketulenan sebatian-sebatian tersebut. Spektrum EDAX menunjukkan tidak terdapat bendasing di dalam sebatian-sebatian tersebut. Fakta ini ditentusahkan lagi dengan kewujudan fasa tunggal seperti ditunjukkan oleh XRD. Disebabkan jisim atom litium yang rendah ia tidak dikesan, melainkan dengan menggunakan EDAX mengesahkan kehadiran atom-atom yang berkenaan. Taburan saiz zarah bagi sebatian-sebatian tersebut menunjukkan bahawa zarah-zarah berkenaan tersebar di dalam isipadu yang besar. Diameter zarah bertambah dengan pertambahan suhu 'kalsinas'. Proses licikan boleh mengurangkan taburan isipadu yang besar dan diameter zarah. Selepas licikan dilakukan dengan mortar dan "pestle hand grinder", saiz zarah menurun dengan ketara dan taburan isipadu dikurangkan.

SEM juga mengesahkan zarah-zarah tersebut adalah bersaiz sub-mikron. Sebatian-sebatian tersebut telah digunakan sebagai bahan katod bagi bateri litium-ion. Analisa ke atas bateri menunjukkan nilai kapasiti bagi LiMn_2O_4 adalah 10 mAh, bagi LiCoO_2 adalah 24 mAh dan bagi $\text{Li}_2\text{Ni}_8\text{O}_{10}$ adalah 5 mAh.

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"Verily never will Allah change the condition of peoples until they change it themselves (with their own souls)" - Al-Quran

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I certify that an Examination Committee met on 18th August 2000 to conduct the final examination of Muhammad Amin Idrees on his Doctor of Philosophy thesis entitled "Preparation and Characterization of Lithium-Based Solid State Battery Materials" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations, which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.



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TABLE OF CONTENT

	Page
ABSTRACT	ii
ABSTRAK	iv
ACKNOWLEDGEMENTS	vi
APPROVAL SHEETS	vii
DECLARATION FORM	ix
LIST OF TABLES	xiii
LIST OF FIGURES	xix
LIST OF ABBREVIATIONS	xxvii
CHAPTER	
I INTRODUCTION	1
II LITIRATURE REVIEW	12
Introduction	12
Types of existing rechargeable batteries for commercial Interest	12
Why lithium batteries?	15
Primary Lithium Batteries	19
Secondary Lithium Metal Batteries	19
Disadvantages of Secondary Lithium Batteries	20
Li-ion batteries or Rocking-chair batteries	21
What is meant by intercalation	25
Intercalation in Graphite	26
Advantage of using Graphite/carbon Anode	28
A New Anode Material for Lithium Secondary Batteries..	29
Selection criteria for intercalation material	30
Types of cathode material	32
Why Lithium Transition Metal Oxides?	33
Lithium insertion Cathode Material	36
Manganese Oxides.	38
Other Oxide	39
III MATERIALS AND METHODS.	41
Introduction	41
Chemicals used	41
Acids used	41
Synthesis procedure	42



	Experimental techniques used	44
	X-ray diffraction	44
	Differential thermal analysis (DTA)	48
	Thermogravimetric analysis (TGA)	52
	Particle size distribution	54
	Energy disperse analysis (EDAX)	57
	Scanning electron microscopy (SEM)	58
	Battery Assembly	60
	Electrolyte preparation	60
	Battery Fabrication	60
	Schematic representation of the fabricated cell	61
	Bttery Charge and Discharge	62
	Characteristics of batteries	64
IV	SAMPLE, COMPOSITION AND SAMPLE PREPARATION	65
	Introduction	65
	Section A (LiMn_2O_4)	67
	Synthesis procedure for poly crystalline LiMn_2O_4 powders by sol-gel method	68
	LiMn_2O_4 at different stages of preparation	70
	Section B (LiCoO_2)	71
	LiCoO_2 at different stages of preparation	74
	Section C ($\text{Li}_2\text{Ni}_8\text{O}_{10}$)	75
	$\text{Li}_2\text{Ni}_8\text{O}_{10}$ at different stages of preparations	77
V	EXPERIMENTAL RESULTS AND DISCUSSION ...	78
	Introduction	78
	Experimental Results and Discussion of LiMn_2O_4	79
	X-ray diffraction analysis	79
	Differetial thermal analysis (DTA)	85
	Thermogravimetric analysis (TGA)	92
	EDAX studies	98
	Particle size distribution	99
	Scanning electron microscopy (SEM) discussion. ...	104
	Battery characteristics	109
	Experimental Results and Discussion of LiCoO_2	114
	X-ray diffraction analysis	114
	Differential thermal analysis (DTA)	120
	Thermogravimetric analysis (TGA)	127
	Energy disperses analysis (EDAX)	132
	Particle size distribution	134
	Scanning electron microscopy (SEM)	138
	Battery characteristics	143

Experimental results and discussion of $\text{Li}_2\text{Ni}_8\text{O}_{10}$	147
X-ray diffraction analysis	147
Differential thermal analysis (DTA)	152
Thermogravimetric analysis (TGA)	159
Energy dispersive analysis (EDAX)	164
Particle size distribution	166
Scanning electron microscopy (SEM)	171
Battery characteristics	176
Summary	179
CONCLUSION	181
Suggestions for future work	185
REFERENCES	186
APPENDICES	194
A Definitions of some Terms	194
B Standard Reduction Potentials of Electrodes Reactions	198
C Electrochemical Equivalents of Battery Materials	200
D Conductivity Ranges of Various Electrolytes	202
VITA	203

LIST OF TABLES

Table		Page
1	Cathode, anode and electrolyte requirements for a good cell (David Linden, 1984)	5
2	Comparison of various batteries, their energy density and the cost	15
3	Types of cathode materials (Prabaharan et al, 1997)	32
4	Formation temperatures of (a-h) routes for LiMn_2O_4 gel precursor powders, by using different starting reagents and acids such as citric or maleic or succinic or tartaric acid as gelating agents	87
5	Formation temperatures of (a-h) routes for LiCoO_2 gel-precursor powders, with different starting reagents and acids such as citric or maleic or succinic or tartaric acid as gelating agents	122
6	Formation temperatures of $\text{Li}_2\text{Ni}_8\text{O}_{10}$ for (a-h) routes, by taking ne starting reagents The molar ratio of citric or maleic or succinic or tartaric acid (gelating agent) to total metal ions was 1 5	159

LIST OF FIGURES

Figure		Page
1	Basic components of the electrochemical cell (Booklet energy information center, 1995)	4
2	Characteristics discharge curves (Linden, 1984)	9
3	Relationship between the equivalent weight (anode plus cathode) and the theoretical specific energy on weight basis of various electrochemical systems (Gabano, 1983)	18
4	Lithium intercalation mechanism (Armand et al, 1980)	22
5	(a) Structure of stage 1, 2 and 3 compounds and (b) In-plane structure of LiC_6 (Dahn et al, 1994)	27
6	Approximate voltages of transition metal (di) oxides in non-aqueous lithium cells Relation between the operating voltages and the number of d-electrons can be seen (Ohzuku et al, 1997)	35
7	The experimental method for the preparation of the final compounds (In-Hwan Oh et al, 1997)	42
8	X-ray diffraction patterns of the gel-derived material calcined at 650°C at the various molar ratios of maleic acid to total metal ions of (a) 0.75, (b) 1.0, (c) 1.5 and (d) 2.0 (In-Hwan Oh et al, 1997)	47
9	Schematic diagram of a DTA apparatus (Pope and Judd, 1977)	49
10	Differential thermal analysis of the gel precursor at an air flow rate of $40\text{ cm}^3\text{ min}^{-1}$ and a heating rate of 5°C min^{-1} (In-Hwan Oh et al, 1997)	50
11	Thermogravimetric analysis for (a) the mixture of metal acetate (Li Mn acetate = 1 : 2, (b) sole adipic acid, and (c) the gel precursor pretreated in a vacuum dryer at 100°C prior to thermal analysis in air at the heating rate of 5°C min^{-1} (Yun-Sung Lee et al, 1998)	53

12	The particle size distribution of LiMn_2O_4 when calcined (a) at 50 and (b) at 800 $^{\circ}\text{C}$	55
13	SEM images of the LiCoO_2 powders when calcined (a) at 500 $^{\circ}\text{C}$, (b) at 600 $^{\circ}\text{C}$ and (c) at 700 $^{\circ}\text{C}$ for one hour in air (In-Hwan Oh et al, 1997)	59
14	Schematic representation of the fabricated cell	61
15	It shows charge-discharge curves with the number of cycles for the $\text{Li} / 1 \text{ M LiBF}_4 - \text{n EC/DEC/LiMn}_2\text{O}_4$ cell using LiMn_2O_4 powder calcined at 750 $^{\circ}\text{C}$. Cycling was carried out Galvan statically at constant charge-discharge current density of 1 mA/cm^2 between 3.6 and 4.3 V (Young-Sung Lee et al, 1998)	63
16	Synthesis procedure for polycrystalline LiMn_2O_4 powders by sol-gel method when maleic acid was used as a gelating agent	68
17	LiMn_2O_4 at different stages of preparation (a) solution, (b) precursor powder and (c) Final compound	70
18	LiCoO_2 at different stages of preparation (a) solution, (b) precursor and (c) Final compound	74
19	$\text{Li}_2\text{Ni}_8\text{O}_{10}$ at different stages of preparation (a) solution, (b) precursor powder and (c) Final compound	77
20	X-ray diffraction pattern of the LiMn_2O_4 at the lowest formation Temperature in this study (when calcined at 208 $^{\circ}\text{C}$). The molar of maleic acid (gelating agent) to total metal ions was 1.5	81
21	X-ray diffraction patterns of LiMn_2O_4 for the lowest formation temperature route (f) from Table 4, when calcined (a) at 208 $^{\circ}\text{C}$, (b) at 400 $^{\circ}\text{C}$, (c) 600 $^{\circ}\text{C}$ and (d) 800 $^{\circ}\text{C}$. The molar ratio of maleic acid (gelating agent) to total metal ions was 1.5	82
22	XRD patterns of LiMn_2O_4 reported in the literature from citrate precursor as a function of annealing temperature (Prabaharan et al, 1995)	83
23	XRD patterns of LiMn_2O_4 reported in the literature from oxalate precursor as a function of annealing temperature (Prabaharan et al, 1995)	84

24(a,b)	Differential thermal analysis for LiMn_2O_4 of (a,b) routes from Table 4, at the stage of gel precursor powder.	88
24(c,d)	Differential thermal analysis for LiMn_2O_4 of (c,d) routes from Table 4, at the stage of gel precursor powder.	89
24(e,f)	Differential thermal analysis for LiMn_2O_4 of (e,f) routes from Table 4, at the stage of gel precursor powder.	90
24(g,h)	Differential thermal analysis for LiMn_2O_4 of (g,h) routes from Table 4, at the stage of gel precursor powder.	91
25(a,b)	Thermogravimetric analysis of (a,b) routes from table 4, gel precursor powders of LiMn_2O_4 at a heating rate of $10^\circ\text{C min}^{-1}$	
25(c,d)	Thermogravimetric analysis of (c,d) routes from table 4, For the gel precursor powders of LiMn_2O_4 at a heating rate of $10^\circ\text{C min}^{-1}$	
25(e,f)	Thermogravimetric analysis of (e,f) routes from table 4, for the gel- precursor powders of LiMn_2O_4 at a heating rate of $10^\circ\text{C min}^{-1}$	96
25(g,h)	Thermogravimetric analysis of (g,h) routes from table 4, for the gel-precursor powders of LiMn_2O_4 at a heating rate of $10^\circ\text{C min}^{-1}$	
26(a)	EDAX spectrum of LiMn_2O_4 sample at 500°C	98
26(b)	EDAX spectrum of LiMn_2O_4 sample at 800°C	99
27(a)	Particle size distribution of a sample LiMn_2O_4 when calcined (a) at 300°C	101
27(b, c)	Particle size distribution of LiMn_2O_4 sample when calcined (b) at 500°C and (c) at 800°C	102
32(a,b)	Particle size distribution of LiMn_2O_4 sample after ground when calcined at 800°C	103
29	Dependence of specific surface area of LiMn_2O_4 , obtained at the lowest formation temperature route (f) from Table 4 when it was calcined at various temperatures.	103

30(a,b)	SEM images for LiMn_2O_4 powder at the lowest formation temperature route (f) from Table 4, at 500 magnification When calcined (a) at $500\text{ }^\circ\text{C}$ and (b) at $800\text{ }^\circ\text{C}$	105
30(c,d)	SEM images for LiMn_2O_4 powder at the lowest formation temperature route (f) from Table 4, at 1000 magnification When calcined (c) at $500\text{ }^\circ\text{C}$ and (d) at $800\text{ }^\circ\text{C}$	106
30(e,f)	SEM images for LiMn_2O_4 powder at the lowest formation temperature route (f) from Table 4, at 1500 magnification When calcined (e) at $500\text{ }^\circ\text{C}$ and (f) at $800\text{ }^\circ\text{C}$	107
30(g,h)	SEM images for LiMn_2O_4 powder at the lowest formation temperature route (f) from Table 4, at 2000 magnification When calcined (g) at $500\text{ }^\circ\text{C}$ and (h) at $800\text{ }^\circ\text{C}$	108
31 (a)	First charge/discharge curve of LiMn_2O_4 cell	110
31 (b)	Second charge/discharge curve of LiMn_2O_4 cell	110
31 (c)	Third charge/discharge curve of LiMn_2O_4 cell	111
3 (d)	Forth charge/discharge curve of LiMn_2O_4 cell	111
31(e)	Fifth charge/discharge curve of LiMn_2O_4 cell	112
32(a,b)	X-ray diffraction pattern of the LiCoO_2 powders calcined (a) at $200\text{ }^\circ\text{C}$ and (b) at $400\text{ }^\circ\text{C}$ The molar ratio of succinic acid (gelating agent) to total metal ion was 1 5	116
32(c,d)	X-ray diffraction pattern of the LiCoO_2 powders calcined (c) at $600\text{ }^\circ\text{C}$ and (b) at $800\text{ }^\circ\text{C}$ The molar ratio of succinic acid (gelating agent) to total metal ion was 1 5	117
33(a-d)	X-ray diffraction pattern of the LiCoO_2 precursor powders When calcined (a) at $200\text{ }^\circ\text{C}$,(b) $400\text{ }^\circ\text{C}$, (c) at $600\text{ }^\circ\text{C}$ and (d) at $800\text{ }^\circ\text{C}$ The molar ratio of succinic acid(gelating agent) to total metal ions was 1 5	118
34(a,d)	From literature, calcined at various temperatures (a) at $500\text{ }^\circ\text{C}$,(b) $600\text{ }^\circ\text{C}$, (c) at $650\text{ }^\circ\text{C}$ and (d) at $700\text{ }^\circ\text{C}$ when maleic acid was used as gelating agent (In-Hwan Oh et al, 1997)	119
35(a,b)	Differential thermal analysis for LiCoO_2 of (a,b) routes from Table 5,	123

35(c,d)	Differential thermal analysis for LiCoO_2 of (c,d) routes from Table 5, at the stage of gel precursor powder	124
35(e,f)	Differential thermal analysis for LiCoO_2 of (e,f) routes from Table 5, at the stage of gel precursor powder	125
35(g,h)	Differential thermal analysis for LiCoO_2 of (g,h) routes from Table 5, at the stage of gel precursor powder	126
36(a)	Thermogravimetric analysis for LiCoO_2 of (a) route from Table 5, at the heating rate of $10^\circ\text{C min}^{-1}$	128
36(b,c)	Thermogravimetric analysis for LiCoO_2 of (b,c) routes from Table 5, at the heating rate of $10^\circ\text{C min}^{-1}$	129
36(d,e)	Thermogravimetric analysis for LiCoO_2 of (d,e) routes from Table 5, at the heating rate of $10^\circ\text{C min}^{-1}$	130
36(f,g)	Thermogravimetric analysis for LiCoO_2 of (f,g) routes from Table 5, at the heating rate of $10^\circ\text{C min}^{-1}$	132
36(h)	Thermogravimetric analysis for LiCoO_2 of (h) route from Table 5, at the heating rate of $10^\circ\text{C min}^{-1}$	136
37(a)	EDAX spectrum of LiCoO_2 powder for the lowest formation Temperature route (c) from Table 5 at 500 magnification when it was calcined at 500°C	133
37(b)	EDAX spectrum of LiCoO_2 powder for the lowest formation Temperature route (c) from Table 5 at 500 magnification when it was calcined at 800°C	134
38(a,b)	Particle size distribution of LiCoO_2 sample (a) at 300°C and (b) at 800°C The molar ratio of succinic acid (gelating agent) to total metal ions was 1 5	136
39	Particle size distribution of LiCoO_2 after grinding when sintered at 800°C	137
40	Dependence of the specific surface area of LiCoO_2 with calcination temperature, for the lowest formation temperature route (c) from Table 5 when succinic acid was used as gelating agent	137

41(a,b)	SEM images of LiCoO_2 powders for the lowest formation temperature route (c) from Table 5 at 500 magnification when it was calcined (a) at 500°C and (b) at 800°C	139
41(c,d)	SEM images of LiCoO_2 powders for the lowest formation temperature route (c) from Table 5 at 500 magnification when it was calcined (c) at 500°C and (d) at 800°C	140
41(e,f)	SEM images of LiCoO_2 powders for the lowest formation temperature route (c) from Table 5 at 500 magnification when it was calcined (e) at 500°C and (f) at 800°C	141
41(g,h)	SEM images of LiCoO_2 powders for the lowest formation temperature route (c) from Table 5 at 500 magnification when it was calcined (g) at 500°C and (h) at 800°C	142
42(a)	First charge/discharge curve of LiCoO_2 cell	143
42(b)	Second charge/discharge curve of LiCoO_2 cell	144
42(c)	Third charge/discharge curve of LiCoO_2 cell	144
42(d)	Forth charge/discharge curve of LiCoO_2 cell	145
42(e)	Fifth charge/discharge curve of LiCoO_2 cell	145
43(a)	X-ray diffraction pattern of $\text{Li}_2\text{Ni}_8\text{O}_{10}$ for the lowest formation Temperature route (f) from Table 6 (when calcined at 214°C) molar ratio of maleic acid (gelating agent) to total metal ion was 1 5	148
43(b,c)	X-ray diffraction patterns of $\text{Li}_2\text{Ni}_8\text{O}_{10}$ when calcined (b) at 400°C and (c) at 600°C The molar ratio of maleic acid (gelaing agent) to total metal ions was 1 5	149
43(d)	X-ray diffraction pattern of $\text{Li}_2\text{Ni}_8\text{O}_{10}$ when calcined (d) at 800°C The molar ratio of maleic acid (gelaing agent) to total metal ions was 1 5	150
44(a-d)	Shows the comparative X-ray diffraction patterns of the gel-Derived materials of $\text{Li}_2\text{Ni}_8\text{O}_{10}$ when calcined ,(a) at 214°C , (b) at 400°C , (c) at 600°C and (d) at 800°C The molar ratio of maleic acid (gelaing agent) to total metal ions was 1 5	151

45(a,b)	Differential thermal analysis for $\text{Li}_2\text{Ni}_8\text{O}_{10}$ of (a,b) routes from Table 6, at the stage of gel precursor powder	155
45(c,d)	Differential thermal analysis for $\text{Li}_2\text{Ni}_8\text{O}_{10}$ of (c,d) routes from Table 6, at the stage of gel precursor powder	156
45 (e,f)	Differential thermal analysis for $\text{Li}_2\text{Ni}_8\text{O}_{10}$ of (e,f) routes from Table 6, at the stage of gel precursor powder	157
45(g,h)	Differential thermal analysis for $\text{Li}_2\text{Ni}_8\text{O}_{10}$ of (g,h) routes from Table 6, at the stage of gel precursor powder	158
46(a)	Thermogravimetric analysis of $\text{Li}_2\text{Ni}_8\text{O}_{10}$ for (a) route from Table 6, at a heating rate of $10^\circ\text{C min}^{-1}$	160
46(b,c)	Thermogravimetric analysis of $\text{Li}_2\text{Ni}_8\text{O}_{10}$ for (b,c) routes from Table 6, at a heating rate of $10^\circ\text{C min}^{-1}$	161
46 (d,e)	Thermogravimetric analysis of $\text{Li}_2\text{Ni}_8\text{O}_{10}$ for (d,e) routes from Table 6, at a heating rate of $10^\circ\text{C min}^{-1}$	162
46(f,g)	Thermogravimetric analysis of $\text{Li}_2\text{Ni}_8\text{O}_{10}$ for (f,g) routes from Table 6, at a heating rate of $10^\circ\text{C min}^{-1}$	163
46(h)	Thermogravimetric analysis of $\text{Li}_2\text{Ni}_8\text{O}_{10}$ for (h) route from Table 6, at a heating rate of $10^\circ\text{C min}^{-1}$	164
47(a)	EDAX spectrum of $\text{Li}_2\text{Ni}_8\text{O}_{10}$ powder when calcined (a) at 500°C The molar ratio of maleic acid (gelating agent) to total metal ions was 1 5	165
47(b)	EDAX spectrum of $\text{Li}_2\text{Ni}_8\text{O}_{10}$ powder for the lowest formation Temperature route (f) from Table 6 when it was calcined at 800°C	166
48(a,b)	Particle size distribution of $\text{Li}_2\text{Ni}_8\text{O}_{10}$ samples when calcined (a) at 400°C and (b) at 600°C when the molar ratio of maleic acid (gelating agent) to total metal ions was 1 5	168
49(a,b)	Particle size distribution of $\text{Li}_2\text{Ni}_8\text{O}_{10}$ (a) without grinding at 800°C and (b) when grinded after calcination at 800°C	169
50	Dependence of the specific surface area of $\text{Li}_2\text{Ni}_8\text{O}_{10}$ at the various calcination temperatures from the lowest formation route in this study when maleic acid was used as gelating agent and the molar ratio of acid to total metal ions was 1 5	170

51(a,b)	SEM images for $\text{Li}_2\text{Ni}_8\text{O}_{10}$ powder at the lowest formation temperature route (f) from Table 6 at 500 magnification when it was calcined (a) at 500°C and (b) at 800°C	172
51(c,,d)	SEM images for $\text{Li}_2\text{Ni}_8\text{O}_{10}$ powder at the lowest formation temperature route (f) from Table 6 at 1000 magnification when it was calcined (c) at 500°C and (d) at 800°C	173
51(e,f)	SEM images for $\text{Li}_2\text{Ni}_8\text{O}_{10}$ powder at the lowest formation temperature route (f) from Table 6 at 1500 magnification when it was calcined (e) at 500°C and (f) at 800°C	174
51(g,h)	SEM images for $\text{Li}_2\text{Ni}_8\text{O}_{10}$ powder at the lowest formation temperature route (f) from Table 6 at 2000 magnification when it calcined (g) at 500°C and (h) at 800°C	175
52(a)	First charge/discharge profile of a $\text{Li}_2\text{Ni}_8\text{O}_{10}$ cell	176
52(b)	Second charge/discharge profile of a $\text{Li}_2\text{Ni}_8\text{O}_{10}$ cell	177
52(c)	Third charge/discharge profile of a $\text{Li}_2\text{Ni}_8\text{O}_{10}$ cell	177
52(d)	Forth charge/discharge profile of a $\text{Li}_2\text{Ni}_8\text{O}_{10}$ cell	178

LIST OF SYMBOLS AND ABBREVIATIONS/NOTATION

Ah	ampere-hour
Ah/L	ampere-hours per liter
Å	angstrom
A	ampere
α	alpha
Ah	ampere-hour
Ah/kg	ampere-hours per kilogram
β	beta
Cm	centimeter
C	coulomb
Cr	chromium
Co	cobalt
Cl	chlorine
Cu	copper
$\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	cobaltous(II) nitrate hexahydrate
$\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$	citric acid
$\text{C}_4\text{H}_6\text{O}_4$	succinic acid
$\text{CH}(\text{COOH})(\text{OH})\text{COOH}$	maleic acid
$(\text{CHOH} \cdot \text{COOH})_2$	tartaric acid
DEC	diethyl Carbonate
DTA	differential thermal analysis
DMC	dimethyl carbonate

E_0	standard potential (in volts)
EC	ethylene Carbonate
EPDM	ethylene propylene diene monomer
F	faraday constant (96500 C or 26.8 Ah)
Fe	iron
g	gram
ΔG	Gibbs free energy change
I	current
IR	Internal resistance of a cell
JCPDS	Standards, Joint Committee on Powder Diffraction Standards, Index to the Powder Diffraction File, Swarthmore, Pa.
Kg	kilogram
Li-ion	lithium-ion
LiNO_3	lithium nitrate
Li_2CO_3	lithium carbonate
$\text{Li}_2\text{Ni}_8\text{O}_{10}$	lithium nickel oxide
LiCoO_2	lithium cobalt oxide
LiMn_2O_4	lithium manganese oxide
LiClO_4	lithium perchlorate
LiMeS_2	lithium transition metal sulphide
LiMeSe_2	lithium transition metal selenide
LiMeO_2	lithium transition metal oxide
LiMO_2	lithium metal oxide

LiFeO ₂	lithium iron oxide
LiBF ₄	lithium-tetrafluoroborat
LiCF ₃ SO ₃	lithium trifluoromethane-sulfonate
LiTi ₂ O ₄	lithium titanium oxide
LiV ₂ O ₄	lithium vanadateoxide
LiCoVO ₄	lithium cobalt vanadateoxide
LiNiVO ₄	lithium nickel vanadateoxide
mAh	milli-ampere hour
m ²	square meter
m ³	cubic meter
M(mole)	amount of substance
Me	transition metal
mAh/g	milli-ampere hour per gram
Mn	manganese
Mo	molybdenum
MCFC	molten carbonate fuel cells
“n”	no of electrons involved in the electrochemical reaction
N	nitrogen
Ni	nickel
Ni-Cd	nickel cadmium
Ni(NO ₃) ₂ · 6 H ₂ O	nickel (II) nitrate hexahydrate
O	oxygen
PVDF	polyvinylidene fluoride